

# PRECISION CLEANING VERIFICATION OF FLUID COMPONENTS BY AIR/WATER IMPINGEMENT AND TOTAL CARBON ANALYSIS

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## ABSTRACT

NASA personnel at Kennedy Space Center's Material Science Laboratory have developed new environmentally sound precision cleaning and verification techniques for systems and components found at the center. This technology is required to replace existing methods traditionally employing CFC-113. The new patent-pending technique of precision cleaning verification is for large components of cryogenic fluid systems. These are stainless steel, sand cast valve bodies with internal surface areas ranging from 0.2 to 0.9 m<sup>2</sup>. Extrapolation of this technique to components of even larger sizes (by orders of magnitude) is planned. Currently, the verification process is completely manual.

In the new technique, a high velocity, low volume water stream impacts the part to be verified. This process is referred to as Breathing Air/Water Impingement and forms the basis for the Impingement Verification System (IVS). The system is unique in that a gas stream is used to accelerate the water droplets to high speeds. Water is injected into the gas stream in a small, continuous amount. The air/water mixture is then passed through a converging/diverging nozzle where the gas is accelerated to supersonic velocities. These droplets impart sufficient energy to the precision cleaned surface to place non-volatile residue (NVR) contaminants into suspension in the water.

The sample water is collected and its NVR level is determined by total organic carbon (TOC) analysis at 880°C. The TOC, in ppm carbon, is used to establish the NVR level. A correlation between the present gravimetric CFC-113 NVR and the IVS NVR is found from experimental sensitivity factors measured for various contaminants. The sensitivity has the units of ppm of carbon per mg/ft<sup>2</sup> of contaminant. In this paper, the equipment is described and data are presented showing the development of the sensitivity factors from a test set including four NVRs impinged from witness plates of 0.05 to 0.75 m<sup>2</sup>.

## INTRODUCTION

In the past, a CFC-113 rinse was used to verify that small fittings, valves and regulators, large valves, pipes, flex hoses and tubing met a non-volatile residue (NVR) contamination specification of less than 11.1 mg/m<sup>2</sup> (1 mg/ft<sup>2</sup>). This NVR verification test was performed on precision-cleaned critical parts, e.g., those used with liquid oxygen service. Previous to the present work with larger parts, small parts NVR verification had successfully been met by the use of water and ultrasonic baths (e.g., Allen, 1993). KSC experiments during 1992-93 identified a Breathing Air/Water (BAir/Water) impingement technique that justified additional development for cleaning verification of large components at KSC. The insolubility of NVR materials in water would be overcome by imparting high shear from an air water jet to suspend the NVR in the collected water. Dearing (1993) and Melton (1993) reported an Impingement Verification System and results of testing to optimize the performance of the air-water impingement technique. Optimum NVR test parameters were: 32 mL/min water flow, 14 L/s (30 SCFM) breathing air flow, 2.06 MPa (300 psig) air-water driving pressure, and 2-min. impingement time for 0.09 m<sup>2</sup> (1 ft<sup>2</sup>) flat plates. In testing the 0.09 m<sup>2</sup> plate, the TOC content of the sample, resulting from ultrasonic impingement with the air-water mixture, was found to be linear with pre-known contaminant level. The TOC was also linear with the NVR removed, the latter determined by CFC-113 rinse after air-water impingement. These results were found for each of five typical fluids and greases used at KSC. Another linear result was observed using a contaminant mixture of eleven greases and fluids. The sensitivities of the various TOC results from known initial contaminant levels were in the range of 1

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to 5 ppm TOC/mg contaminant. Contaminant removal efficiencies for the optimum conditions were on the order on 40%, as proven by a subsequent CFC-113 rinse.

Melton (1994) introduced an empirical equation to relate measured TOC to pre-known contamination level. The results were correlated such that an average sensitivity,  $S$ , was determined. The empirical equation then permitted a calculation of the equivalent NVR (ENVR) based on a measurement of TOC from impingement samples. The ENVR is the value which would have resulted from a CFC-113 rinse and gravimetric NVR measurement:

$$\text{ENVR} = \frac{V_s * (\text{TOC}_s - \text{TOC}_b)}{V_{av} * S * A^2}, \text{ mg/ft}^2 \quad (1)$$

where  $V_s$ =actual collected volume (mL), TOC = total organic carbon for impingement (s) or blank (b) in ppm,  $V_{av}$ =average volume of sample based on duration of impingement and water flow rate (e.g., 45 mL for the valves),  $S$ =sensitivity, ppm net TOC/(mg-ft<sup>2</sup>) and  $A$ =area of impinged surface. (Note that originally the exponent of  $A$  was 1.0.) Calibration experiments were required to determine  $S$ . The procedure was to contaminate a clean valve with a known amount of NVR, and then impinge the surface and measure the net TOC. The above equation was then used to calculate  $S$  by substituting the known contaminant amount per unit area of interior surface. Although the data were widely scattered, the results did show a promising correlation for valves. The average sensitivity was 3.4 ppm/mg for three contaminants: Amoco Rykon II (hydrocarbon grease), Chevron Molykote (molybdenum disulfide grease), DC-55M (Dow-Corning silicone grease), and Mil-H-83282 (hydraulic fluid).

This report describes further development of a new precision-cleaning verification method which employs a supersonic jet of air and water drops instead of CFCs. The report includes data collected and analyzed in a series of tests to calibrate the method with known contamination amounts placed on flat witness plates. The project scope includes witness plates from 0.05 to 0.75 m<sup>2</sup> (0.5 to 8 ft<sup>2</sup>), using one hydraulic fluid and three common lubricants as contaminants, each at levels of 11.1 and 22.2 mg/m<sup>2</sup> (1 and 2 mg/ft<sup>2</sup>)

## INSTRUMENTATION AND PROCEDURE

Details of the impingement apparatus and procedure were presented in Melton (1993 and 1994). Briefly, the equipment consists of a regulated gas supply, a pressurized water tank, a water metering and injection device, a flex hose, air-water mixer tee, nozzle assembly, catchpan, and associated valves, fittings, and hardware. A contaminant is applied to a witness plate with a swab which is differentially weighed. A flex hose attached to a wand with a supersonic nozzle is moved slowly and repeatedly over the plate surface for a specified time (below), thus spraying the air-water mixture onto the surface. Water drips off the surface and is caught in a beaker. The water sample volume is measured and several 200 microliter samples are injected in the TOC instrument which operates at 880°C. (Inorganic carbon is checked and found to be small and roughly constant.)

## RESULTS

Figure 1 shows calculated values of ENVR vs. the plate area, which is also proportional to the contaminant amount (only greases shown). The horizontal dashed lines show the range of values for contaminant levels of 11.1 and 22.2 mg/m<sup>2</sup>. Despite the scatter, there is a clear distinction between ENVRs for these levels. A key result is the dependency of  $S$  on the different contaminants and the amount (or area). Figure 2 indicates that there is excessive scatter in  $S$  values at or below  $A=0.09$  m<sup>2</sup> (1 ft<sup>2</sup>). Otherwise,  $S$  values vs. plate area are reasonably close and may be averaged. An equation which fits  $S$  vs.  $A$  is:

$$S = a (A)^b \quad (2)$$

Examples of values of a and b are:

Contam / Level	Am Ryk	Am Ryk	Chev Moly	Chev Moly	DC-55M	DC-55M	Mil-H	Mil-H
mg/ft <sup>2</sup>	1.0	2.0	1.0	2.0	1.0	2.0	1.0	2.0
a	2.86	1.68	5.17	5.53	5.19	5.15	5.14	5.56
b	-1.26	-1.39	-1.38	-1.43	-1.41	-1.40	-1.43	-1.42

Once S values are known, the measurement scheme is the following: 1) Estimate surface area and impinge part for an appropriate time; 2) collect water and inject sample (and blank) into TOC apparatus; 3) calculate S from Eqn. 2; measure volume of water collected and calculate average volume from the product of water flow rate and impingement time; 5) substitute all variables into Eqn. 1. Times are selected by a tradeoff between accuracy and convenience. The times used in this work were  $t = \sqrt{(2A)}$ , min., where A is in square feet.

Some cautions are appropriate: 1) The values of S calculated from Eqn. 2 will depend on the expected contaminant types, surface shape, spraying time, operator technique, etc. Once a set of values of S is measured, a conservative average S should be adopted so that some clean plates may be failed, but no dirty plates are passed; 2) there is a strong interference, giving high TOCs, from isopropyl alcohol and other volatile organics, food vapors, etc., which are used nearby or infiltrate the air ducts; 3) to apply this method to another specific set of contaminants and surfaces, calibrations must be performed starting with known contaminant types and amounts; 4) contaminants with little or no carbon, e.g., Krytox 240AC fluorocarbon, are not detectable with TOC analysis.

## CONCLUSIONS

The Breathing Air/Water Impingement technique using the Impingement Verification System has proven to be consistent and useful in verifying lubricant contaminant levels on witness plates. This method works well with a known contaminant, or a limited group. Further work is required to make the IVS more generally applicable to unknown contaminants, and to define its limitations.

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FIG. 1 ENVR VS. PLATE AREA

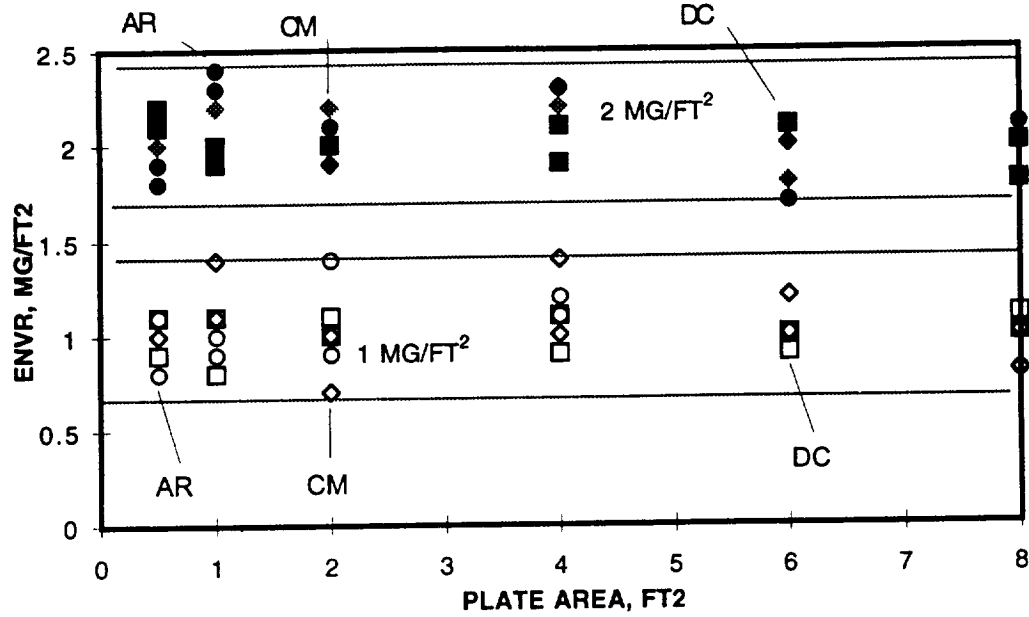


FIG. 2 SENSITIVITY VS. PLATE AREA

